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13. ABSTRACT (Maximum 200 words)

This report describes the development of thermal analysis instrumentation for studies of the kinetics of chemical reactions and phase transformations of shock compressed powder materials. The instrumentation consists of a Perkin-Elmer differential thermal analyzer and a "power-compensated" differential scanning calorimeter, both equipped with controlled gas flow and vacuum. The objective of the instrumentation is to accurately determine the thermodynamics and kinetics of chemical and structural changes occurring in shock-compressed materials. The thermal analysis system is used to obtain fundamental data, including onset temperatures for chemical reactions, amorphous-to-crystalline phase transformations, and austenite/martensite transformation temperatures in shock densified powder mixtures. The measurements allow determination of the apparent activation energies, to model the enhanced reaction and transformation behavior of shock-compressed materials.

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THERMAL ANALYSIS INSTRUMENTATION FOR KINETICS OF SHOCKED MATERIALS

FINAL REPORT

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1. INTRODUCTION

The unusual combination of high pressure, large strain, and high deformation rates produced during shock compression of powders, can lead to extensive plastic deformation and heating, particle comminution, fluid-like turbulent flow, and mixing of constituents with fresh and cleansed surfaces.[1,2] These effects significantly alter the mechanical, physical, structural, and chemical characteristics of the starting materials, thereby enhancing the solid-state chemical reactivity and generating an highly-activated dense-packed state of material. Such a state of material can result in significantly altered kinetics of chemical or structural changes, causing reactions or phase transitions to occur during microsecond duration of shock compression,[1-7] as well as in time-scales of thermal equilibration following shock-compression, or even during subsequent (post-shock) thermal treatments in reduced time scales or at substantially lower temperatures.

Synthesis of materials based on fabrication of shock-densified and highly activated state of material followed by post-shock thermal treatments, is a process that holds much promise. Past studies have been focused on documenting the enhanced sinterability of otherwise difficult-to-sinter super-hard ceramic powders.[8,9] The approach has also been applied to (i) reactive powder mixtures to form compounds at temperatures significantly below the melting point of reactants,[10-12] (ii) precipitation-strengthened materials to increase the number density of precipitate nucleation sites during subsequent aging,[13] and (iii) recrystallization (de-vitrification) of amorphous compounds to form single-phase nanocrystalline structures with inhibited grain growth.[14] The kinetics of such structural and chemical changes has been studied using post-mortem microstructural analysis of the shock-compressed and reacted materials, by quantifying the fraction of phase formed as a function of time and temperature, from which the kinetics is established assuming Arhenius or other kinetics models.[15] Thermal analysis using DTA and DSC can be used to obtain precise information about reaction initiation conditions and activation energy values independent of an assumed reaction-kinetics models. Thermodynamic data, e.g., enthalpy of reaction/transformation, can also be obtained. The thermodynamic and kinetic data can then be used as (i) measure of the degree of activation in shock-compressed materials and (ii) to deduce the mechanism and optimal processing conditions for post-shock thermal treatments for materials synthesis applications.

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In this program, instrumentation for thermal analysis of shock-compressed materials has been developed and used to obtain thermodynamic and kinetic properties as measures of the degree of shock activation and optimization of post-shock materials synthesis conditions. The equipment includes a Perkin-Elmer high-temperature DTA capable for use at temperatures up to 1650°C, and a precision "power-compensated" DSC for measurements of transformation enthalpy. A gas flow and atmosphere-control system has also been developed. A detailed description of the thermal analysis instrumentation developed is provided in Section 2. The thermal analysis instrumentation developed is being used to support the various projects for our on-going research supported by ARO under Grant No. DAAG55-97-1-0163 entitled "Synthesis of Intermetallic-Ceramic Composites with Ultra-fine Microstructures." Some of the results obtained with the newly developed instrumentation are described in Section 3.

2. DESCRIPTION OF THERMAL ANALYSIS INSTRUMENTATION

Thermal analysis provides a valuable tool for monitoring the thermodynamics and kinetics of structural phase transformations and chemical reactions in materials as a function of temperature. In particular, the differences in the kinetics of structural and chemical changes in shock-compressed and unshocked materials can be established and used to optimize the processing conditions required to synthesize compounds with desired microstructural characteristics and properties. The Differential Thermal Analyzer (DTA) measures the difference in temperature between a sample (undergoing transformation/reaction) and a reference material, both of which are exposed to the same heating schedule via their symmetric placement with respect to the furnace. The reference material remains inert and undergoes no phase/chemical change upon heating through the temperature range of interest. The temperature difference between the sample and reference is measured by a "differential" thermocouple in which one junction is in contact with the underside of the reference crucible. When the sample undergoes a structural or chemical change, thermal energy is either absorbed (e.g., during melting) or released (e.g., during solidification, crystallization, reaction in elemental powders). The DTA instrument indicates the transformation by showing "endothermic" (with negative amplitude) or "exothermic" (with positive amplitude) peaks corresponding to the temperature differential arising from the sample being cooler or hotter than the reference due to the phase change.[16]

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The operating principle of the differential scanning calorimeter (DSC) is totally different from that of the DTA.[16] The output traces are, however, virtually similar, and are plotted as heat flow (or energy released) as a function of temperature or time. The DSC has separate containers for both the sample and the reference, which also have their individual heating elements and temperature measuring devices. The sample and the reference chambers are heated equally through the temperature regime of the transformation. As the sample temperature infinitesimally deviates from the reference temperature, the device detects it and reduces the heat input to one cell while adding heat to the other so as to maintain a zero temperature differential between the sample and reference, thereby establishing a "null balance." The quantity of electrical energy per unit time which must be supplied to the heating elements (beyond that required for normal schedule), in order to maintain the null balance, is considered to be proportional to the heat released per unit time by the sample, due to phase transformation. This "null balance" description is the basis of the operating principle of the Perking-Elmer DSC, and is also referred to as the "power-compensated" DSC design.

The thermal analysis instrumentation capability developed in this program includes a differential thermal analyzer (The Perkin Elmer DTA 7) and a differential scanning calorimeter (The PYRIS 1 DSC Lab System). Both systems have a common controller. An atmosphere control attachment has also been built to allow for analysis to be performed under good vacuum or inert-gas flow conditions. The specifications of the two systems are given below.

PYRIS 1 DSC LAB SYSTEM - computer-ready lab system with Pyris 1 DSC operating on the exclusive Perkin Elmer power compensation principle; sample holder consists of independent dual furnaces constructed of Pt-Ir alloy with independently distributed Pt- temperature sensors; unique thermal guard design provides superior baseline reproducibility and unequaled instrument precision; extended temperature range achievable from as low as -170°C to 750°, with cooling rates from 0.1°C/min to 500°C/min, as well as true isothermal operations; temperature precision is 0.01°C; cooling time is under four minutes from 750°C to 100°C; Pyris Series TA manager - containing the 32-bit thermal analysis applications software library, which runs under Windows 95, is provided for control, data acquisition, and analysis.

DTA 7 LAB SYSTEM - high-temperature quantitative testing up to 1600°C; Boersma DTA design operating in standard DTA mode (measuring and displaying delta T) and heat flux DSC mode (measuring and displaying heat flow); scan rates from 0.1 to 100°C/min; unique cooling design allows rapid cool-down at end of test; use of vacuum and various atmospheres allowed; modular unit features six unique status indicators; TAC 7/DX thermal analysis controller link used with 7 series/UNIX series system; and software library containing routines for instrument control, and data acquisition and analysis.

3.   RESULTS OBTAINED WITH THERMAL ANALYSIS SYSTEM

Materials synthesis based on use of shock activated/modified materials has been considered as a promising concept in processing applications where the enhanced solid-state reactivity influences the subsequent solid-solid, solid-liquid, and solid-gas interactions in single and multiple component systems. Thermal analysis studies have been performed to obtain fundamental information about the enhancement in the kinetics of reaction/transformation behavior due to shock activation/modification. Examples of some of these effects will be described next.

3.1   Reaction Behavior of Shock Densified Powder Mixtures

Self-sustaining exothermic chemical reactions can be initiated in reactive mixtures of elemental powders upon heating above the melting temperature of the low-melting point constituent. The reaction then proceeds by the dissolution of the solid into the molten constituent followed by precipitation of the reaction product. The reactions become self-sustained if the heat of reaction (ΔH_r) is well in excess of the heat of fusion (ΔH_f) needed to melt the material (i.e., $\Delta H_r + \Delta H_f \ll 0$), and the reaction time t_r is much shorter than the time required for the dissipation of the heat released (ΔH_m) into the environment.[17] Under these conditions, the adiabatic reaction temperature often exceeds the melt temperature of products, yielding a material with excess porosity. The porosity results from volume change associated with the difference between densities of reactants and products, expulsion of volatile impurities, and shrinkage due to solidification.[18]

Self-sustained reactions can be inhibited and the reaction made to occur in the solid-state, if the heat dissipation rate is increased, or if the solid-state mass transport (diffusion) rate is accelerated. The latter can be accomplished either by using slow heating rates, or by increasing the diffusivity of reactants due to presence of defects. Bordeaux and Yavari[38] have demonstrated the influence of heating rate on the reaction behavior of Pd-Sn co-laminated composites. Using In differential scanning calorimetry (DSC) experiments, they demonstrated partial occurrence of solid-state diffusion reaction (with peak at ~393K) followed by the main exothermic reaction (with peak at ~510K) occurring with the melting of Sn. They showed that with increasing heating rates, the onset and peak of the solid-state reaction were shifted to higher temperatures, until at heating rates greater than 160 K/min, the solid-state peak was totally inhibited resulting only in the main exothermic reaction that occurs with the peak at ~520K. Alternatively, at lower heating rates, almost complete reaction occurs in the solid-state, leaving no reactants available to release heat at temperatures exceeding the melting point.

Reduction of the reaction onset temperature has also been observed in mechanically ground SiO_2-Al thermite powder mixtures.[19] While unground powder mixtures react at approximately 600°C, the reaction onset temperature in ground powder mixtures decreases with increasing milling time. The enhanced solid state chemical reactivity in ground SiO_2-Al powder mixtures, is attributed to "mechanochemical activation" of the powders. The mechanical milling process involves repeated deformation, fracture, and cold welding of powder particles between colliding balls. The large number of defects introduced in powders, surface cleansing effects, and intimate contacts between nascent surfaces, promote solid-state diffusion and strongly influence the reaction rates.

Shock-compressed powder mixtures also demonstrate such mechanochemical activation effects, resulting in reactions dominated by solid-state defect-enhanced diffusion and occurring at significantly reduced temperatures. However, in highly exothermic powder mixtures, once the reaction is initiated it may become difficult to control the reaction mechanism and rate. If at any instant, the heat released becomes localized and results in local temperature increases exceeding the melt temperature of a reactant, the reaction will be taken over by a self-sustained combustion-type reaction, in spite of reaction initiation in the solid state. Thermal analysis is thus, essential to determine the reaction onset temperatures, degree of reaction occurring in the solid-state, and the amount of heat released during

reaction, in both, un-shocked as well as shock-compressed materials. Thermal analysis can also be used to determine the apparent activation energy as a measure of the degree of shock activation, based on which optimized conditions for thermal treatments can be predicted to ensure that materials synthesis is dominated by a solid-state diffusion reaction.

3.1.1   The Ti-Si System

Reaction kinetics in dynamically-densified Ti-Si powder mixtures, were studied using Differential Thermal Analysis (DTA) in conjunction with XRD measurement of Ti_5Si_3 reaction product formed during heating the compacts in a furnace at temperatures of 800 to 1000°C. Results obtained from DTA analysis showed the presence of two distinct exothermic reactions, corresponding to a lower temperature solid-state diffusion reaction and a higher-temperature self-sustained combustion-type reaction. DTA traces showing the exothermic events as a function of temperature and time, are illustrated in Figure 1 (a) and (b), respectively.

The apparent activation energies for reactions in the dynamically-denisfied Ti-Si powder mixtures were calculated using the modified Kissinger method expressed by the following equation:[20]

$$d[\ln(\frac{H}{T_p^2})]/d(\frac{1}{T_p}) = -\frac{E}{R}$$ (1)

where, H is the heating rate, T_p is the peak temperature, E is the apparent activation energy, and R is the universal gas constant. The activation energy is obtained from the slope of a plot of $ln(H/T_p^2)$ vs $1/T_p$. Accordingly, the apparent activation energy for the lower temperature solid-state diffusion reaction is calculated to be ~ 106 kJ/mol, while the activation energy for the final combustion process (involving dissolution and reprecipitation) is calculated to be ~ 166 kJ/mol. These activation energy values were then used to establish the reaction kinetics based on models for solid-state diffusion and self-sustained combustion-type reactions.
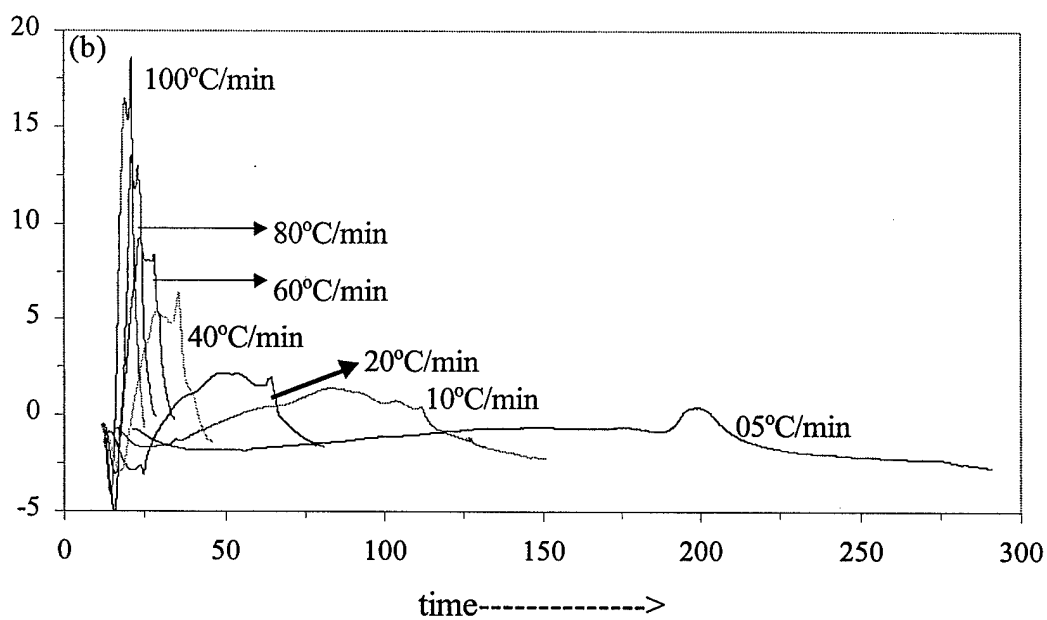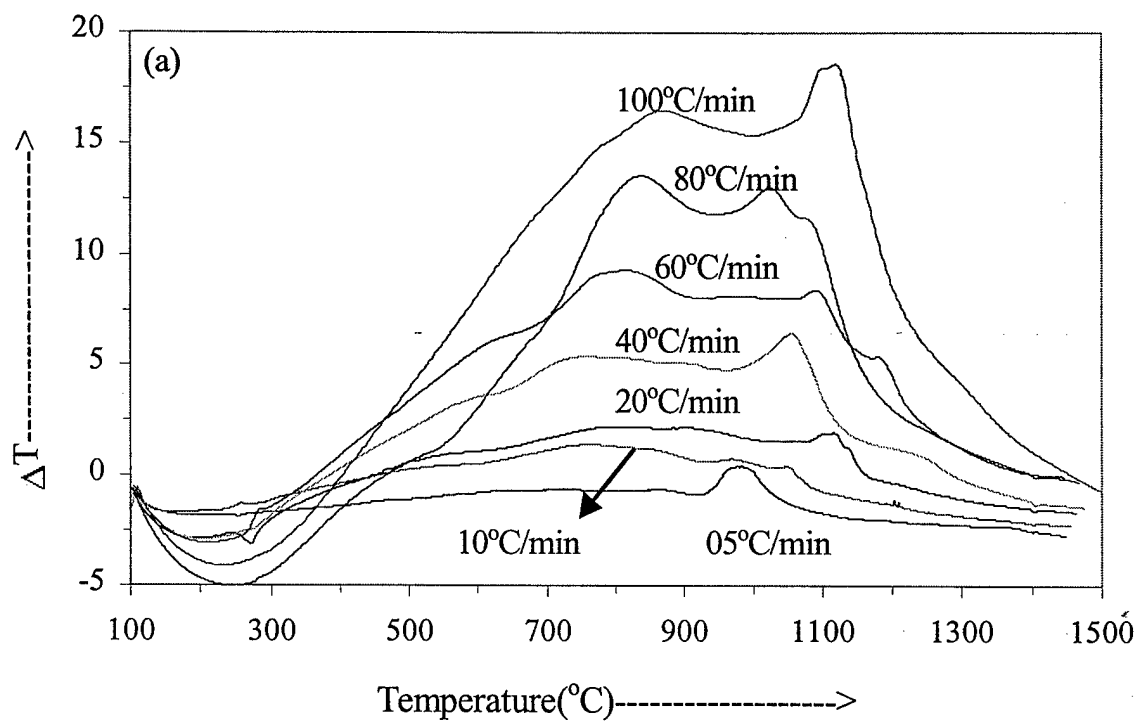
Figure 1.   DTA traces for the shock-densified Ti-Si powder compact at different heating rates, showing: (a) ΔT vs Temperature and (b) ΔT vs time.

The Johnson-Mehl-Avrami (JMA) kinetic model[21] describing the combustion reaction and Carter's model[22] describing solid-state diffusion reactions were considered as the two mechanisms describing the reaction behavior of shock-densified Ti-Si powder mixture compacts. The JMA kinetics model is expressed as:

$$F = 1 - \exp(-kt^n) \qquad (2$$

where F is the fraction transformed, k is a temperature dependant, time independent rate constant, t is the time, and n is the order of the reaction. Carter's solid-state diffusion model, is described using:[21]

$$[1 + (Z-1)F]^{2/3} + (Z-1)(1-F)^{2/3} = [\frac{(1-Z)Dk}{r^2}]t \qquad (3)$$

where, Z is the volume change from reactants to products for a unit amount of reactants. Both D and k are described by an Arrehenius type of equation, $D/k = D_o/k_o \exp(-E_a/RT)$, where Do and k_o are pre-exponential constants and E_a is the activation energy. The activation energies calculated using the modified Kissinger method were used in the above equations, and the fraction of reaction products, F, was calculated as a function of time for various temperatures and k_o values. The D_o value used for the Ti-Si system was 8.1 x 10^{-2} cm²/sec.[22] These calculated results were compared to the reaction fractions, obtained via quantitative x-ray diffraction analysis of samples synthesized in the furnace at various temperatures and hold times for a sample shock-densified at 5 GPa. Figure 2 shows a plot of F vs $ln(t)$ for the experimental data and the best fits to this data for the above two models. The k_o values for the best fits for JMA and Carter's kinetics were 1.95 x 10^3 and 1.85 respectively. It can be seen that at 800 and 900°C, there is poor co-relation between the JMA kinetics and the experimental data, but a very good match with Carter's solid-state reaction kinetics model for the entire reaction stage. In other words, even with up to 70% product formation the rate of heat released is less than that dissipated, hence the solid-state reaction is not taken over by the combustion process at these temperatures. However, with an increase in temperature to 1000°C, the results show the experimental data approaching the JMA model, within a hold time of less than an hour. Thus, at a furnace temperature of 1000°C, the dynamically-densified Ti-Si powder mixture compacts react initially (up to 50% product formation) via a solid-state mechanism, and the remaining reaction occurs in the combustion mode via the dissolution and reprecipitation mechanism. The results demonstrate that dynamic densification can

activate powder mixtures and lower the apparent activation energy. Thus, iit is possible that the degree of reaction occurring in the solid state or via the combustion mode can be controlled to tailor the reaction synthesis process.
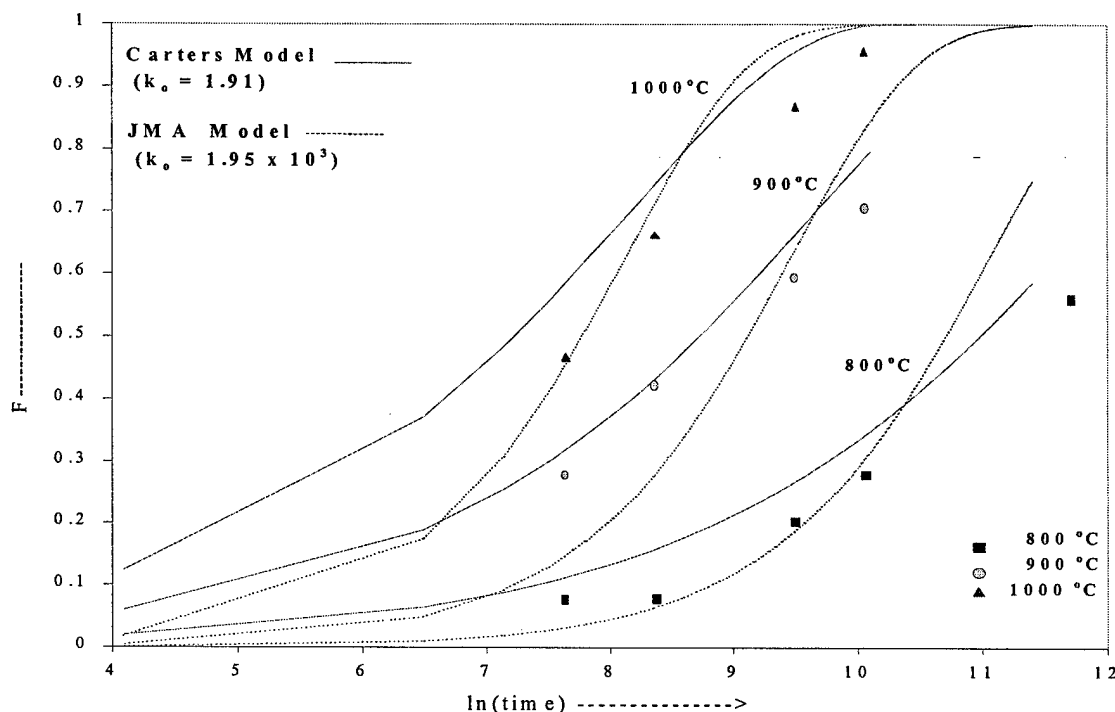

Figure 2: Experimental data of fraction reacted versus time at different temperatures and its comparison to Carter's solid-state and Johnson-Mehl-Avrami reaction kinetics models.

3.1.2 Mo + 2 Si powder compacts

Differential Thermal Analysis (DTA) was performed on dynamically-densified compacts of Mo + 2 Si powder mixtures at different heating rates, to investigate the influence of shock activation on the reaction behavior of this highly exothermic system. The DTA analysis was performed on ~15 mg samples, obtained from a 95% dense compact. Figure 1 displays the DTA scans of Mo + 2Si compacts at heating rates of 10, 40, 70, and 100°C/min. It can be seen that the onset temperatures at all heating rates are the same, however, at the lowest heating rate of 10°C/min, very little reaction is observed until the melting temperature of silicon (1414°C) is reached upon which an exothermic peak is seen

indicating the occurrence of a self-sustained combustion-type of reaction. As the heating rate is increased, a wide peak with increasing area at high heating rates appears to evolve prior to melting of silicon and the accompanying sharp exotherm corresponding to the self-sustained reaction. This broad peak is attributed to the heat released at a rate slower than that of heat dissipation. The amount of reaction (evident by the area under the curve) is greatest in highest heating-rate case (100°C/min), because at high heating rates, the heat released due to reaction at a local area is sufficient to trigger reactions in adjacent areas due to heat localization effects, but is not quite enough to lead to a catastrophic combustion-type reaction until at temperatures exceeding the melting-point of silicon. In summary, it can be seen that differential thermal analysis of Mo + 2 Si densified compacts, allows the characterization of the type of reaction and temperature, or temperature range, over which they occur.
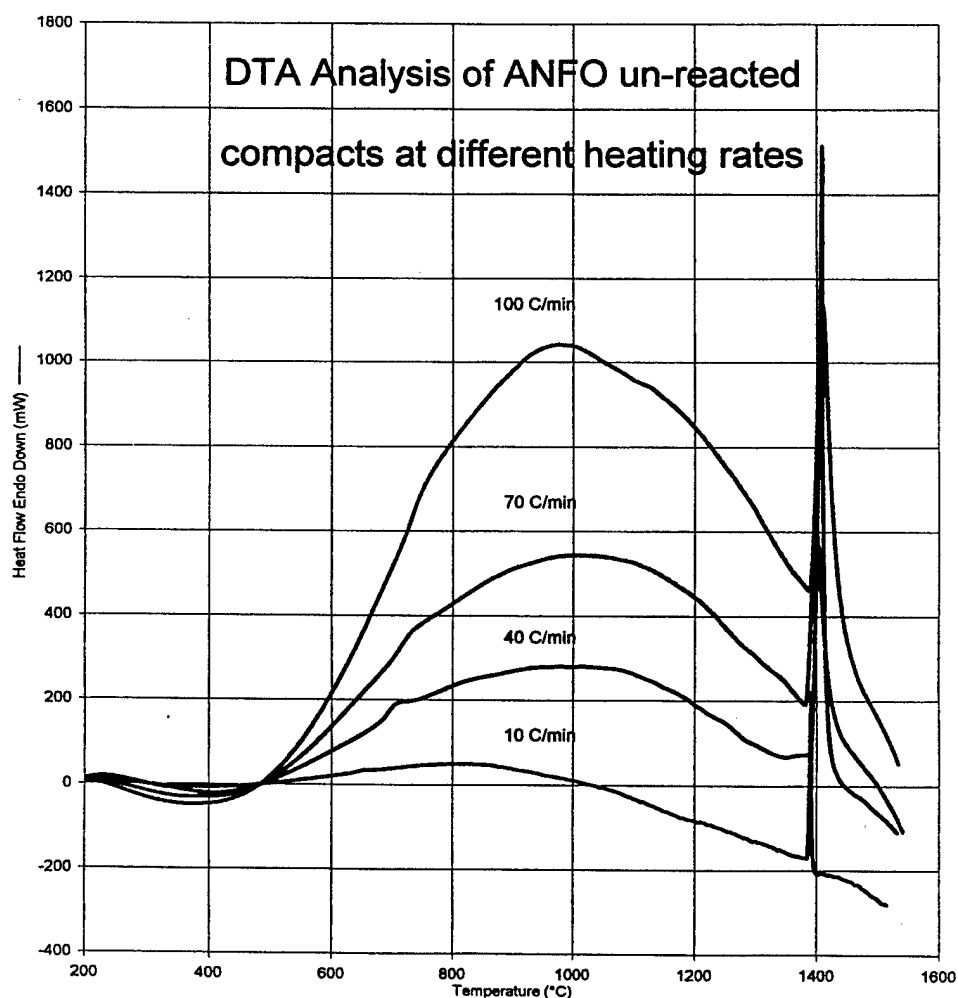

Figure 3.   DTA scans of Mo + 2Si compacts at heating rates of 10, 40, 70, and 100°C/min.

3.2   Crystallization Behavior of Shock Compressed Alloys and Nanomaterial Synthesis

Our preliminary studies have involved the investigation of the crystallization behavior of shock-compressed mechanically amorphized Ti-Si alloy powders possible syntheis of bulk nanocrystalline solids.[14] Elemental titanium (Ti) and silicon (Si) powders of ~10 μm average particle size, were blended in a 5Ti:3Si molar ratio and mechanically amorphized using a Spex 8000 ball mill for 24 hours. Shock densification using a 3-capsule plate-impact fixture was used to produce 10 mm diameter by 3 mm thick compacts. Compacts made at 300 m/s impact velocity (P = 2.5 GPa bulk and 3.5-4.5 GPa axis) were 82-84% dense, while those made at 500 m/s impact velocity (P = 6-8 GPa bulk and 12-15 axis) were 95-99% dense. The mechanically amorphized Ti-Si powders and samples obtained from the compacts were annealed at 600° to 1200°C for varying times (1, 3, and 12 hours). Based on XRD analysis it was observed that the ball-milled powder undergoes primary crystallization to Ti_5Si_3 and secondary crystallization to the Si-rich $TiSi_2$ compound (~10wt.% based on intensity ratios of corresponding peaks). In contrast, the shock-densified material showed polymorphous crystallization to a single-phase Ti_5Si3 compound, with no trace of $TiSi_2$. The difference in the crystallization behavior of the shocked material was attributed to the influence of shock pressure in lowering the free energy of the Ti_5Si3 phase relative to the Si-rich compound. Consequently, it undergoes polymorphous crystallization forming single phase Ti_5Si_3. The average Ti_5Si_3 crystallite size in the crystallized shock-densified compacts, obtained from XRD line-broadening analysis, was observed to be stable at 80-90nm at temperatures as high as 1200°C. Microhardness measurements made on these crystallized shock densified compacts were observed to be in the range of 1100-1200 kg/mm^2 in samples annealed at up to 800°C and between 1300-1400 kg/mm^2 in samples annealed at 1000-1200°C. It was established that defects introduced during shock compression, provide the nucleation sites for crystallite formation during subsequent annealing and also limit the growth of existing crystallites due to impingement, thereby restricting grain growth.

In the present work, the crystallization behavior of shape memory NiTi alloy, and its transformation temperatures in compacts with nanocrystalline grain size, were determined using DSC and DTA analysis. Nitinol powder (-325 mesh, Special Metals) was ball-milled to mechanically amorphize it prior to shock compaction. The crystallization of the amorphous powder was characterized by DTA. The crystallization temperature was found to be ~347°C, and the crystallization energy was

observed to increase with ball-milling time, indicating that the degree of amorphization increased with increasing ball-milling time, as shown in Figure 4.
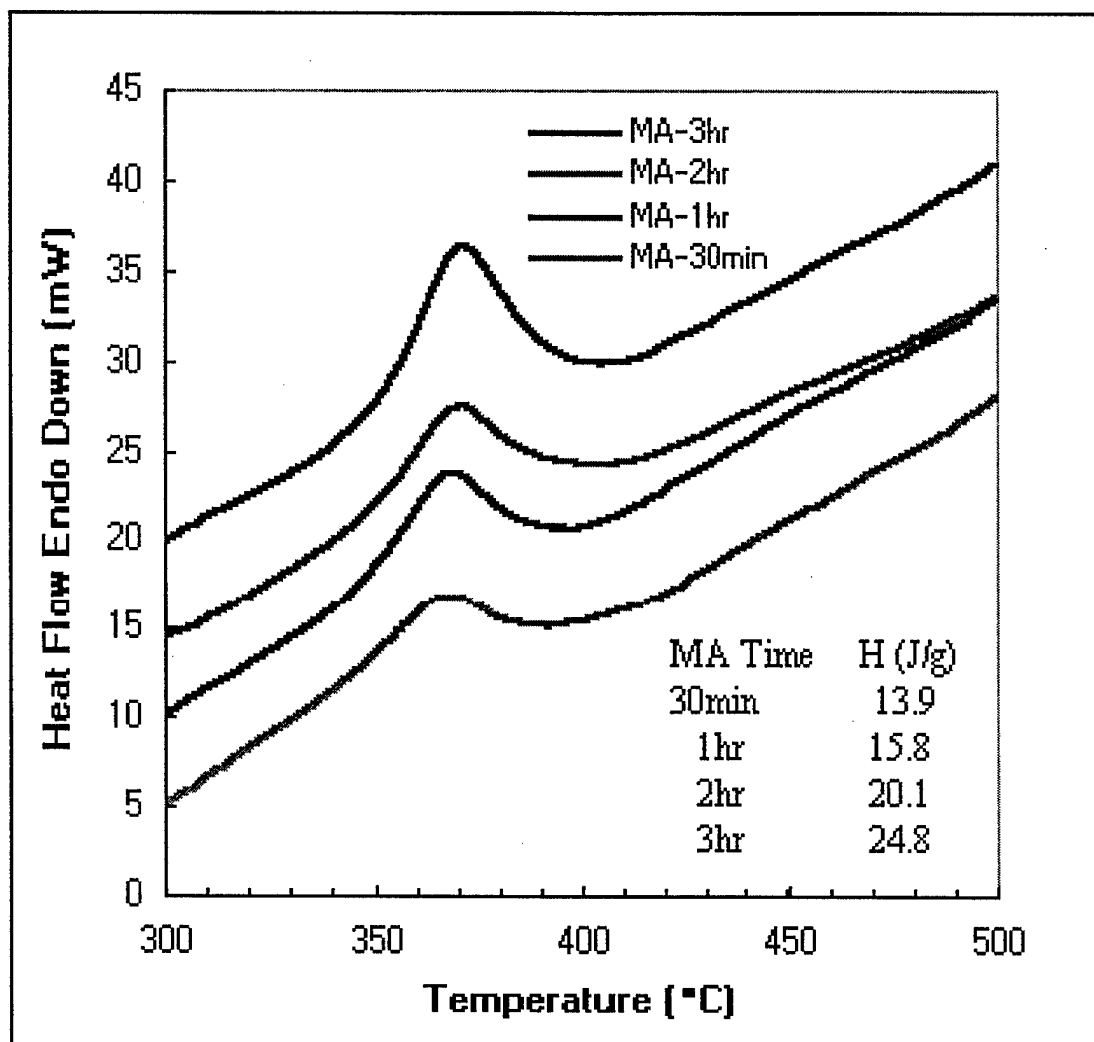

Figure 4.   DTA traces of mechanically-amorphized and shock densified powder compacts and showing increasing enthalpy of crystallization, with increasing ball milling time.

DSC analysis was performed on the (a) as-received Nitinol powder (Figure 5), (b) mechanically amorphized powder ball-milled for various times and subsequently crystallized (Figure 6), and (c) shock-densified mechanically-amorphized powder following crystallization (Figure 7). The results demonstrate that in general, the M_s temperature increases as the grain size reduces to nano-range. The various transformation temperatures are listed in Table I below.
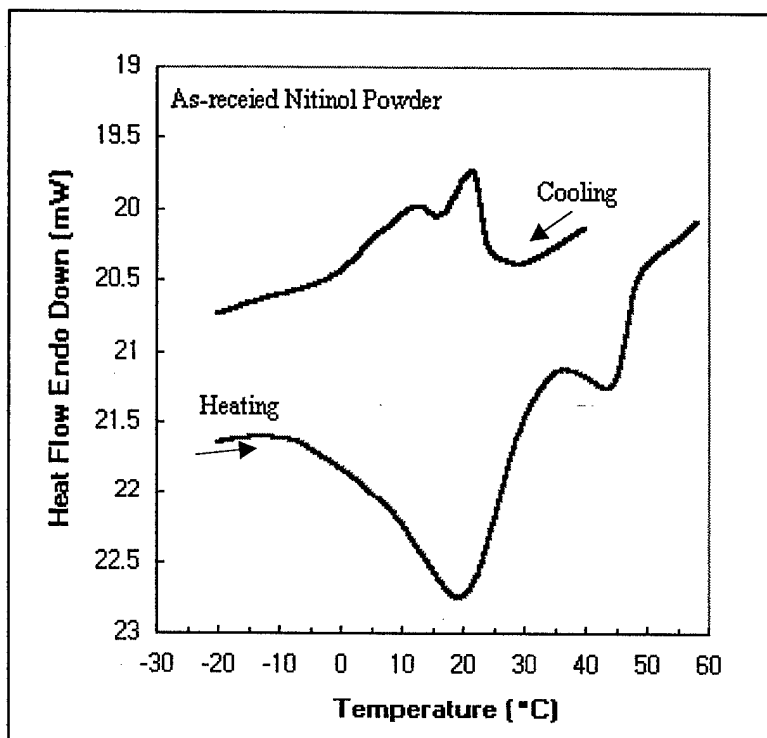
Figure 5.   DSC trace of as-received Nitinol powder during heating and cooling cycles.
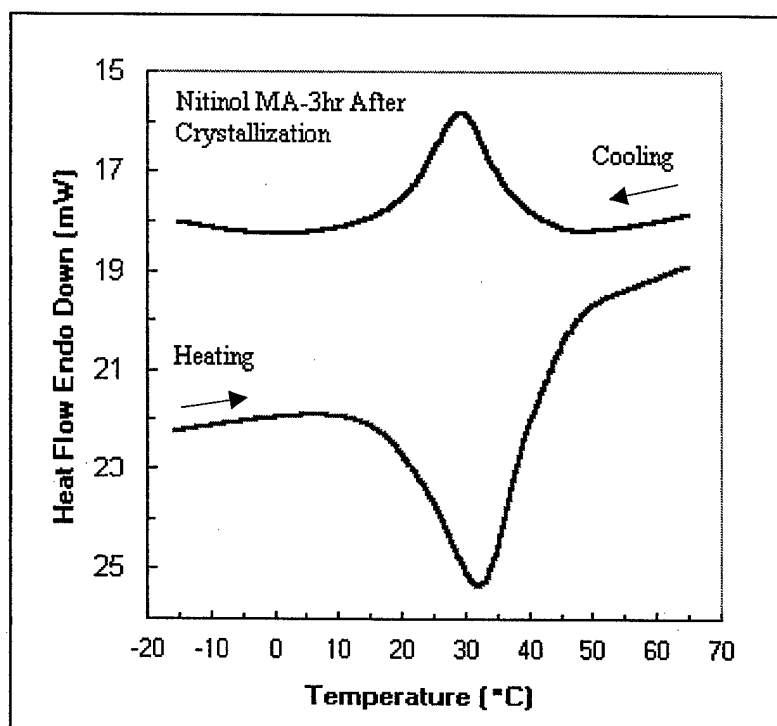

Figure 6.   DSC trace of Mechanically-amorphized (3hr) and recrystallized powder
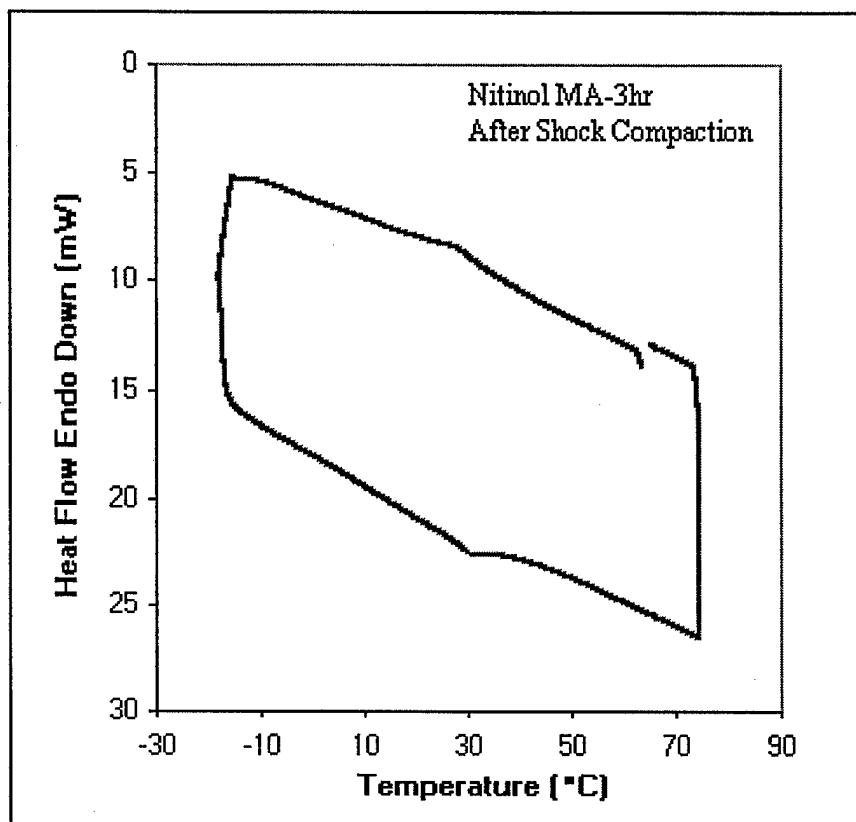
Figure 7. DSC trace of mechanically-amorphized and shock-compacted sample following crystallization.

TABLE I - B2-TO-MONOCLINIC MARTENSITIC TRANSFORMATION TEMPERATURES

Temperature (°C)	M_s	M_f	Peak	A_s	A_f	Peak
Nitinol as-received	28	1	24	7	36	23
MA-3hr	47	30	40	31	49	43
MA-10hr	44	7	22	12	30	24
As-received compact	41	19	30	18	44	33
MA-3hr compact	36	19	28	27	41	32
MA-10hr compact	...	...	...	53	133	88

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